

Viscometer for the Rapid Determination of Viscosities at a Single Concentration

In the chromatographic fractionation of high polymers either by adsorption¹ or precipitation extraction,² there is a need for the viscosity analysis of large numbers of small samples. Using the equations presented by Hart³ for polystyrene, polymethyl methacrylate, or cellulose acetate in specific solvents, the intrinsic viscosity can be determined from the measurement of the specific viscosity at a single concentration. Once the calibrations have been performed, Hart's method should be applicable to the single concentration determination of the intrinsic viscosities of other polymers. On this basis it would appear that a viscometer designed specifically for the rapid determination of single concentration viscosities of successive samples would have a general value.

With the usual Ostwald type of viscometer, samples of the same volume must be pipetted into the viscometer. Although the suspended level type of viscometer eliminates

the need for identical sample volumes, a common requirement of both types of viscometers is the need for rinsing, recalibrating, and drying between measurements on different samples, a procedure which is both time consuming and laborious. The single concentration viscometer described herein incorporates the suspended level principle but eliminates the necessity of rinsing and drying between samples (see Fig. 1).

The design achieves nearly complete drainage by having the solution return through a straight tube, the 2 mm. capillary tube, into a removable solution reservoir which constitutes the sample tube. It will be noted that a medium length joint is used on the viscom-

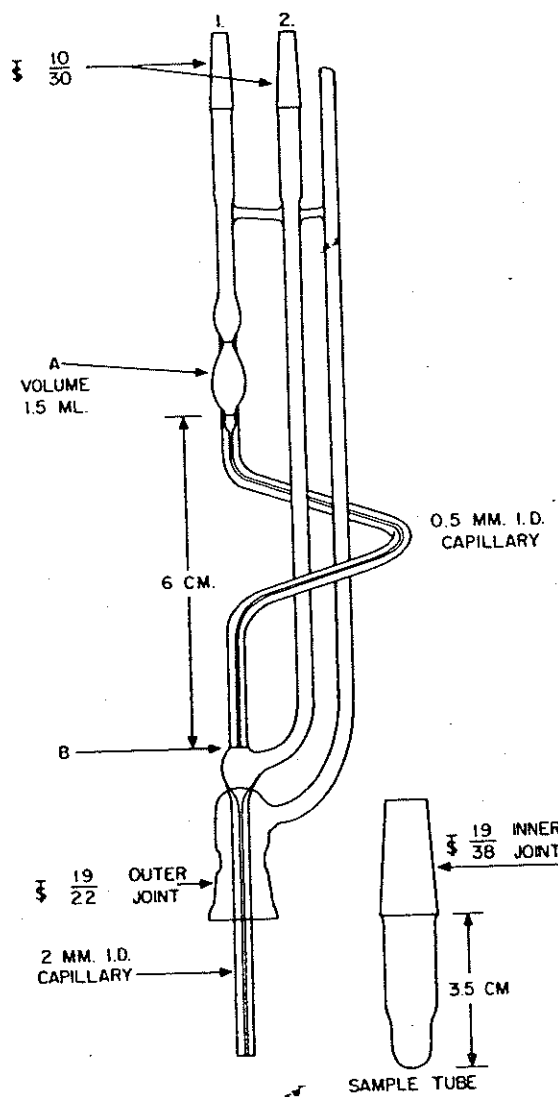


Fig. 1. Diagram of viscometer.

eter while the sample tube carries a full length joint. This restricts the greased area to the lower half of the inner joint on the sample tube and, thus, eliminates any possible contamination of solution by the grease. To demonstrate that the cross contamination of successive samples was negligible, measurements were made on a solution of polystyrene in toluene having a flow time of 350 seconds. Then, without rinsing, a sample tube containing 3 cc. toluene was inserted into the viscometer. Although the flow time difference between the solution and the solvent was 227 seconds, the flow time of the toluene was increased by only 1.6 seconds. Since the maximum difference in the flow time of successive fractionation samples is less than 20 seconds, the error due to cross contamination

would be less than 0.2 second which is equivalent to the absolute timing error in viscosity measurements.

In a usual run, from six to twelve polymer samples are centrifuged in stainless steel tubes in the #40 rotor of the Spinco preparative ultracentrifuge. Three- to five-milliliter samples are pipetted from the centrifuge tubes into individual sample tubes. After these tubes are greased lightly, they are plugged directly into the viscometer. In subsequent steps, the operation of the viscometer is similar to that of the Ubbelohde viscometer. Tube 2 is closed while the solution is drawn into tube 1 to fill the measuring bulb at A. When tube 2 is opened, the liquid clears from the bulb at B at the base of the capillary, isolating the flowing column of liquid, A to B, from the reservoir. Thus, the flow time is independent of the sample volume. After three measurements of flow time, the viscometer is removed from the bath and wiped off. The sample tube is removed and the drop of solution in the tip of the 2-mm capillary is shaken out. A new sample tube can then be plugged in place.

Some of the constants of the viscometer shown are: capillary radius 0.25 mm.; capillary length about 16 cm.; toluene flow time at 30°C. 123 seconds; overall height 29 cm. The sample volume required is somewhat less than 3 cc.

References

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